KWOC
Spruce Budworm —
New Brunswick

A CHEMICAL RESIDUE SURVEY IN RELATION TO THE 1980 SPRUCE BUDWORM SPRAY PROGRAM IN NEW BRUNSWICK

-A Pilot Project-

by

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Note: To facilitate comprehension of this report the reader is urged to obtain from the appropriate agencies original copies of the maps and schemes shown herein.

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SUMMARY

This report contains data resulting from the analysis for fenitrothion in a total of 465 water and air samples in relation with the 1980 spruce budworm spray program in New Brunswick. Water sampling was done in the Nipisiguit River near Bathurst (three sites) and two brooks, namely Location 44 and Little Elbow River (two sites) feeding into the Nipisiguit River. Another location along Route 430 near Sevogle Airstrip consisted of a small "beaver pond" (one site). Water was also sampled in the Canaan River and two brooks feeding into, namely, East Brook Canaan and South Brook Canaan (three sites). A third location was the Turtle Creek Reservoir of the City of Moncton (one site).

Air was sampled at three sites, one was at Sevogle Airstrip, another where Route 430 crosses the Nipisiguit River and a third on campus at Université de Moncton.

Fenitrothion in water was usually detected corresponding approximately with spraying in the immediate area of the sites. The maximum that was ever detected was 20.0 ppb and persistence was usually limited to a few days except in the "beaver pond" where fenitrothion was detected for a total of 18 consecutive days. Aminofenitrothion was also detected with a maximum of 8.0 ppb. Pre-spray and post-spray samples did not contain

any trace of either fenitrothion or aminofenitrothion.

Fenitrothion was only detected occasionally in air samples the maximum being 1.2 ng/dm^3. However, aminofenitrothion was present in several air samples at a maximum of 12.0 ng/dm^3. Although there is no ambiguity as to the origin of fenitrothion, that of aminofenitrothion is uncertain.

The results indicate that in general the contamination was low in sprayed areas of the Province and practically non-existant in non target areas.

INTRODUCTION

Aerial spraying of insecticides as formulations has been carried out almost annually since 1952 in New Brunswick to control spruce budworm (Choristoneura fumiferana Clemens) infestations. Initially the major insecticide was DDT (dichloro-dibenzotrichloroethane) but its use was abandoned mostly because of persistence in the environment and subsequent accumulation in fatty tissues of many organisms along the food chain. Since 1967 the main substitute has been fenitrothion (0,0-dimethyl 0-(3 methyl-4-nitrophenyl) phosphorothioate) although aminocarb (4-dimethylamino-3-methyl phenol methyl carbamate) has gained some importance in recent years.

Much effort has been made in the past to document the distribution, persistence and breakdown of fenitrothion in the environment. Yule and Duffy in 1972 (1) found that at 4 Oz per acre (210 g/ha), fenitrothion disappeared rapidly from mixed spruce foliage ($t_{\frac{1}{2}}$ = 4.5 days) but persisted in trace amounts up to a year, whereas in soil it disappeared within 32 days. Sundaram (2) studied the persistence of aerially applied fenitrothion in foliage, soil, and water samples. In foliage they found 3.5 to 11.7 ppm of fenitrothion half of which was lost within 2 days and 95% in 10 days. After 155 days there was still 0.005 ppm remaining adsorbed in cuticular waxes. The oxon was found only in traces for a short period and nitrocresol was not detected. In soil they found

less than 0.005 to 0.100 ppm of fenitrothion which disappeared within 45 days. No breakdown product was detected. In stream water they found 25.5 - 9 ppb of fenitrothion with $t_{\frac{1}{2}}$ from 0.25 to 3.5 days.

Because of its widespread use particularly in Eastern Canada fenitrothion has been the subject of many evaluations. In 1975 the "Associate Committee on Scientific Criteria for Environmental Quality" sponsored by the National Research Council of Canada held a symposium in Ottawa on the use of fenitrothion and its effects on environmental quality (3). One general conclusion was that scientific criteria for the evaluation of the long-term implications of using fenitrothion could not be established because of a lack of information and several recommendations were made one of which was the need to study the pattern of degradation of fenitrothion in a natural environment.

In New Brunswick, environmental surveillance to measure the effects of spray operations for forest protection against spruce budworm has been on-going since 1976 under the auspices of the New Brunswick Committee for Environmental Monitoring of Forest Insect Control Operations (EMOFICO). In all, four reports has been published by EMOFICO, the latest in 1980 (4).

Many studies carried out by government agencies, universities and private laboratories have been summarized in these reports

and the possible environmental impacts have been discussed.

The Water Quality Branch of Environment Canada in Moncton, N.B. has been actively involved in fenitrothion related studies (ANNEX 1). In 1976 they looked at fenitrothion in soil litter, water and sediments. In 1977 the deposition of fenitrothion was studied using dishes containing Amberlite XAD-2 resin. The adsorption of fenitrothion on sediments and its disappearance in a still water pond were also studied. In 1978 an evaluation of XAD columns versus the conventional solvent extraction procedure for environmental samples was done. Fenitrothion was analysed in rain water using XAD-4 resin. The persistence and vertical distribution of fenitrothion in Peabody Lake was studied and so was the use of fresh water mussels as indicators of fenitrothion contamination of water. Estuarine and lake sediments were sampled for fenitrothion residues. In 1980 the effort was limited to the measurement of off-target fallouts of fenitrothion in two non-target areas, namely, Fundy National Park (N.B.) and CFB Saint Mary (N.B.).

Most of the studies already reported had environmental impact as primary objective and chemical analyses were done with that objective in mind. Another objective was to account for the disappearance of the insecticide in terms of chemical degradation and half-life. Therefore many of the studies involved measurement of chemical deposit under a spray cloud

or following a spray application and the number of samples tested was usually less than one hundred.

The objective of the present study was to assess contamination by fenitrothion across the Province as a result of the spray program to control the spruce budworm. This was to be accomplished by measuring the amounts present at some selected sites for the duration of the spray program, and taking a much larger number of samples (not necessarily in a sprayed area).

Because spray operations were to begin soon and time was running short it was decided this year to limit the survey to the determination of fenitrothion in water and air samples. However the method used (XAD resins and analysis by gas chromatography with a phosphorus specific detector) was also sensitive to the presence of derivatives such as fenitrooxon and aminofenitrothion (5).

SUBSTRATES AND LOCATION OF SITES

Water sampling was carried out in three main areas. The bulk of the sampling was in the Nipisiguit River area, near Bathurst, N.B. where extensive and continued spraying was predicted. The sites on the Nipisiguit River and brooks feeding into consisted of fast running water. In contrast a site was established in a

small beaver pond on Route 430 where the water was shallow and calm (see Figure 1, zones 8 and 11, respectively).

Water sampling in the Canaan River area was located in zone 23, (Figure 1), near Moncton, a localized area where spraying was limited (two sprays in a seven-day span). The turtle Creek area (i.e., the City of Moncton reservoir) was chosen to represent a non-sprayed area.

Air sampling was done at three sites: one in the Nipisiguit River area, the other at Sevogle airstrip not far away, and a third (to act as control) in the City of Moncton. Details regarding location of the sites are given below.

With referecne to Figure 1 as shown, part of the upper area in zone 6 was sprayed with Bt. The extreme part of zone 8 (bottom left) was not sprayed. Zone 31 was deleted from the spray program.

Sampling Sites and Spray Details (refer to Map Booklet available from Librarian and Lands Branch, Dept. of Natural Resources, Fredericton, N.B. or enquire at any Forest Ranger Detachment in N.B.; also Figure 2).

(1) Area I

Sevogle and Nipisiguit River (Northumberland and Gloucester Co., see Figures 3 and 4)

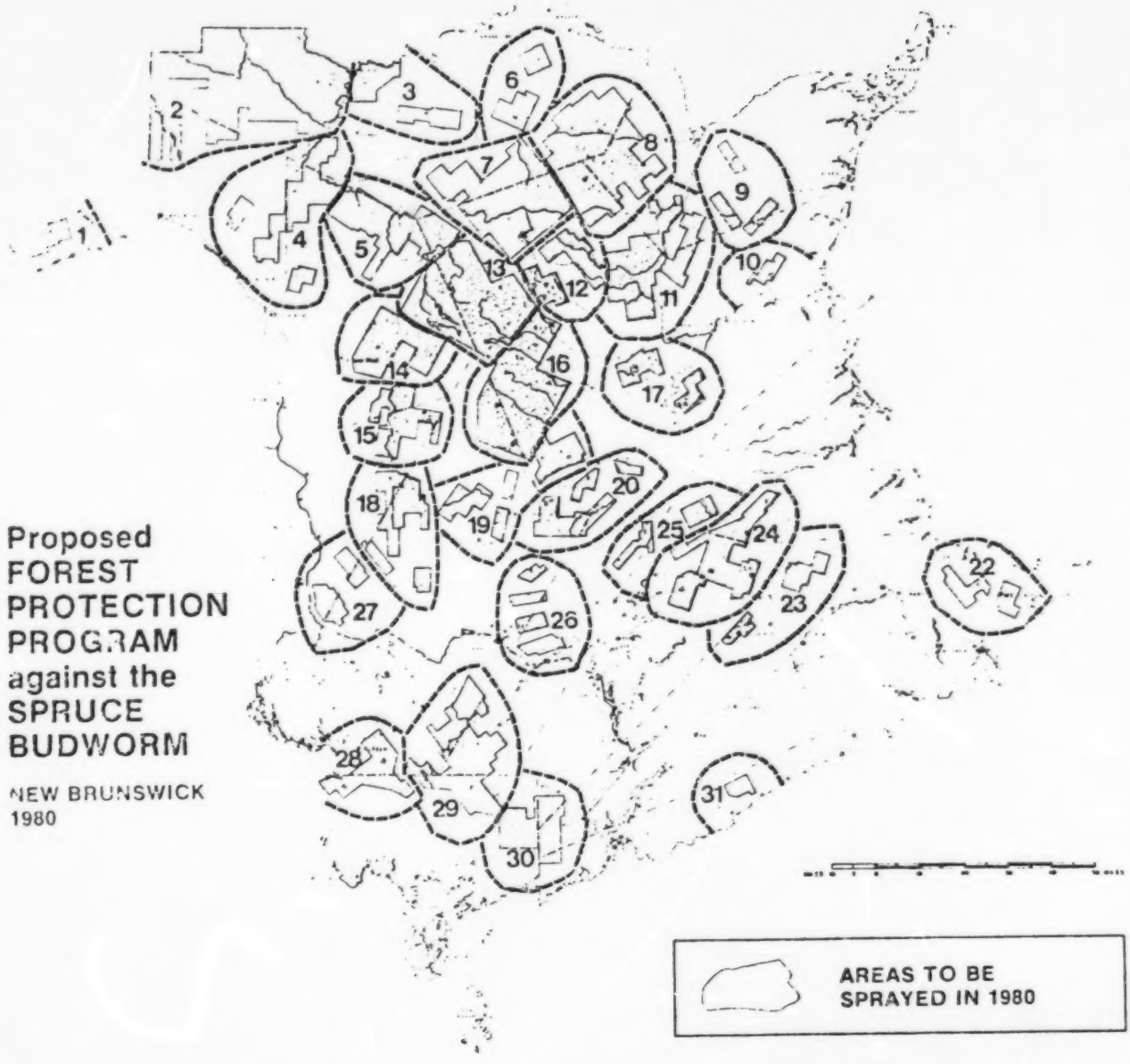
Site I: Sevogle airstrip (0-10), air sampling twice a day

Site II: Nipisiguit River at bridge on Route 430 (0-8), air and water sampling twice a day

Site III: Bathurst Mines Dam on Nipisiguit River (0-7), water sampling once a day

Site IV: Little Elbow River at bridge (M, N-8), water sampling once a day

Figure 1


Proposed
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AREAS TO BE
SPRAYED IN 1980

Figure 2

PROPOSED SPRUCE BUDWORM SPRAY MAP

SHOWING SPRAY BLOCKS BY NUMBER

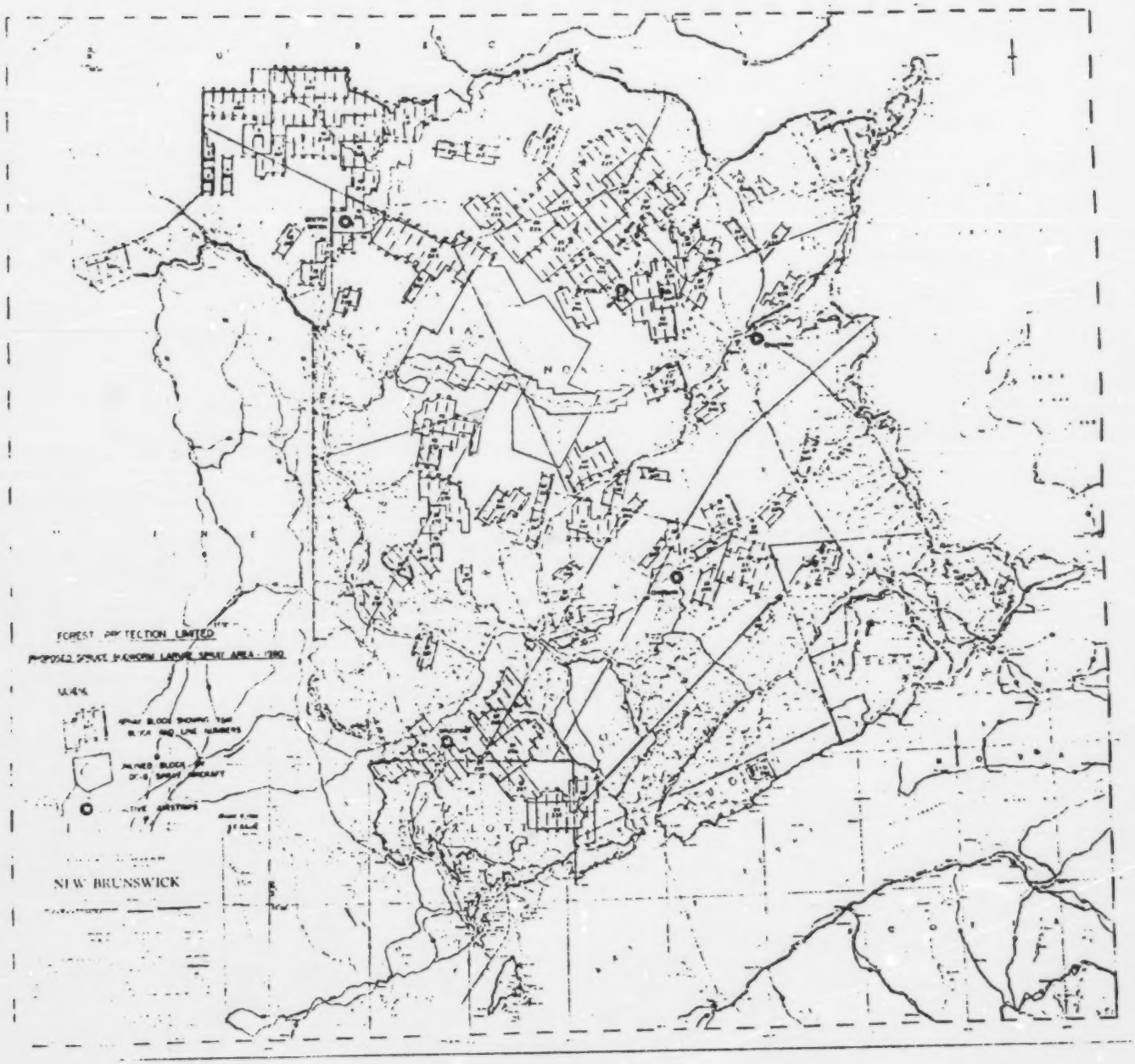

Figure 3

DNR ROAD MAP

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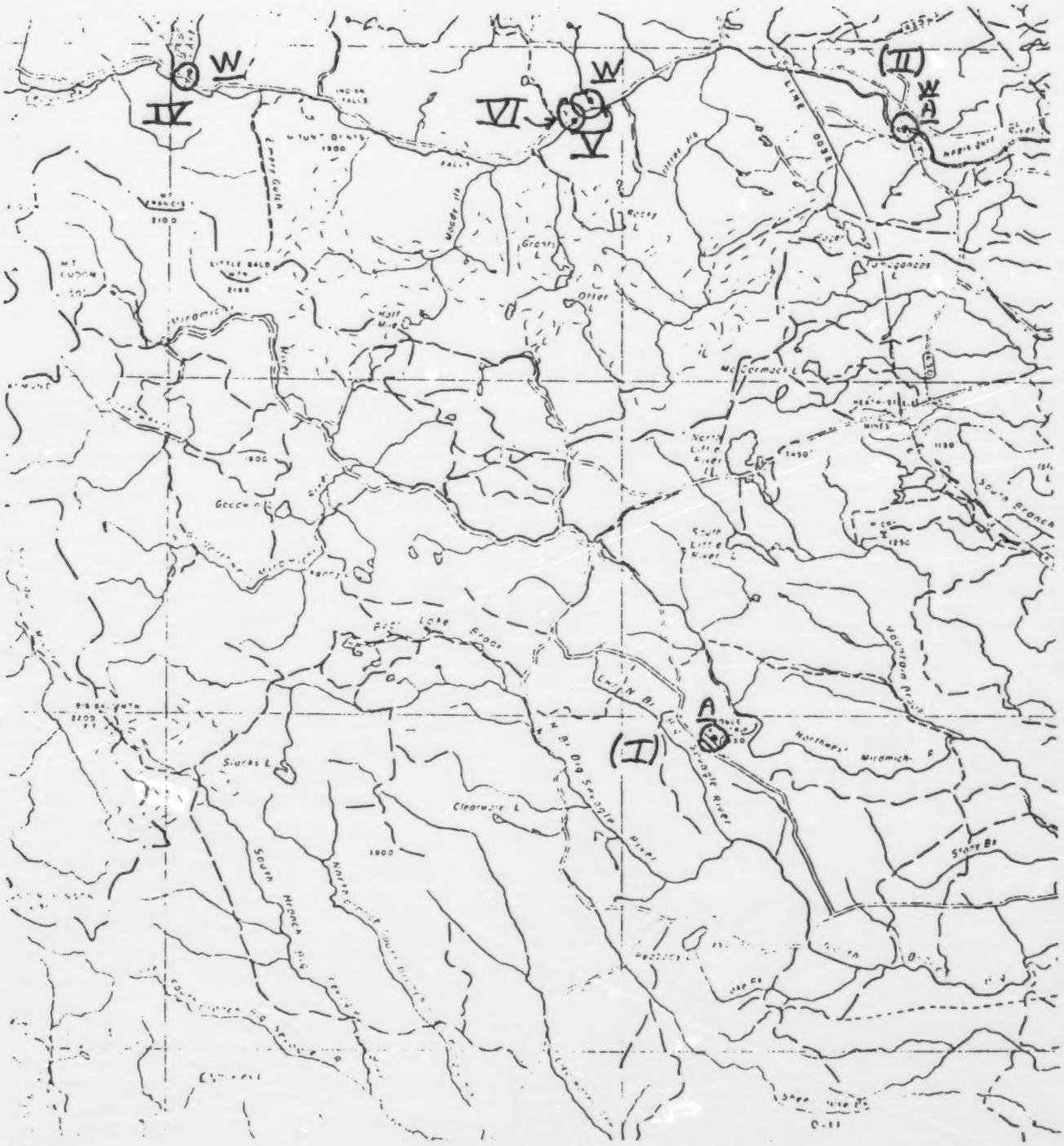
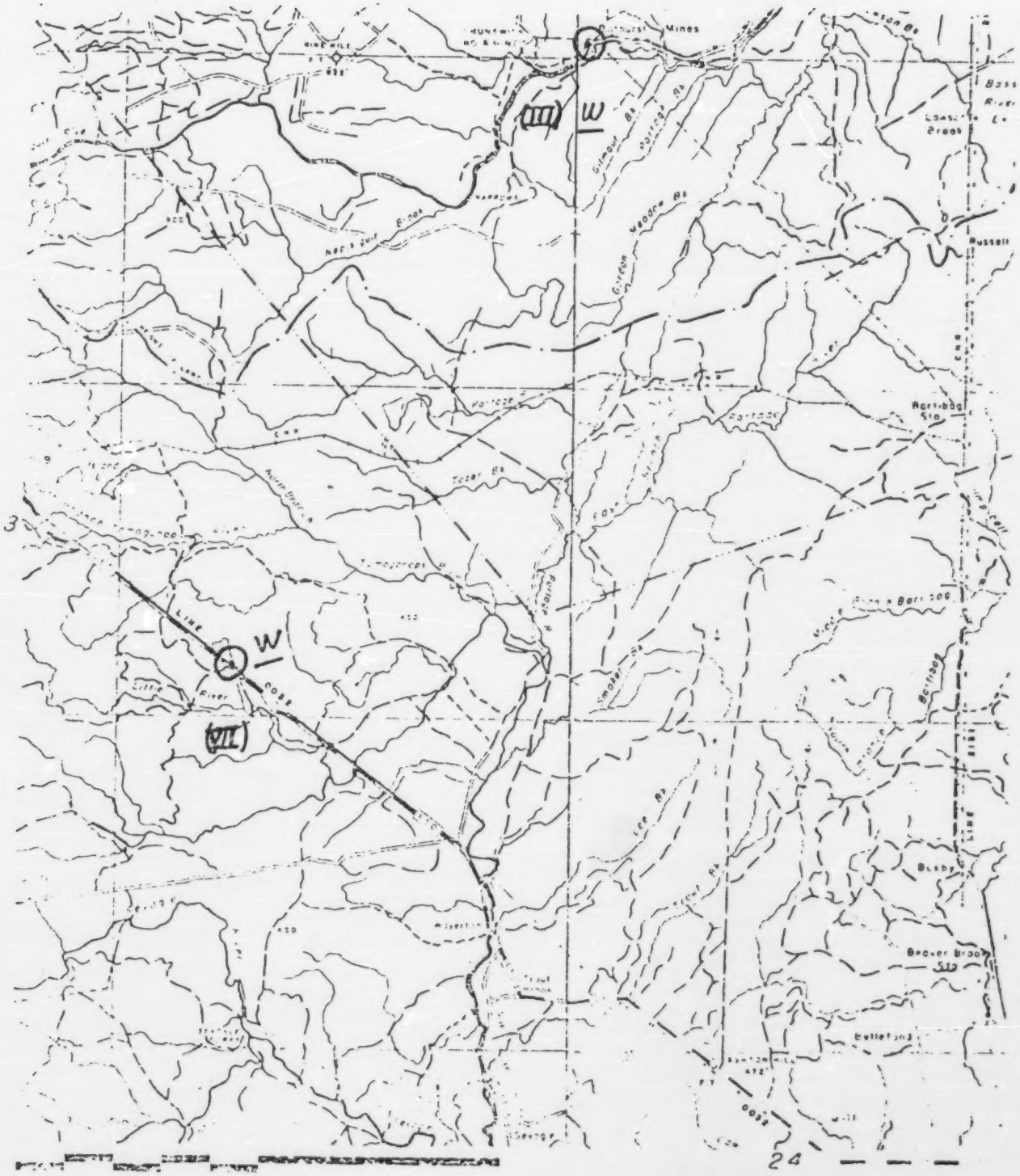


Figure 4

DNR ROAD MAP

Page 19



Site V: named Location 44 by DNR (brook from Three Corner Lake) (N-8), water sampling once a day

Site VI: Nipisiguit River just above Location 44 (N-8), water sampling once a day

Site VII: a beaver dam on Route 430 (P-9), water sampling twice a day

(2) Area II (see Figure 5)

Canaan Games Management area (West. Co.)

Site VIII: at East Canaan on Canaan River at bridge (S-21), water sampling once a day

Site IX: South Brook Canaan River at bridge (T-20), water sampling once a day

Site X: East Brook Canaan River at bridge (T-19), water sampling once a day

(3) Area III (see Figure 5)

Turtle Creek area (West. Co.)

Site XI: Turtle Creek, City of Moncton Reservoir (U-22), at dam, water sampling once a day

(4) Area IV (see Figure 5)

Moncton area (West. Co.)

Site XII: Top of Taillon Building, on Campus of Université de Moncton (U, V-20), air sampling 24h a day

All water samples were picked at the surface (within

Figure 5

DNR ROAD MAP

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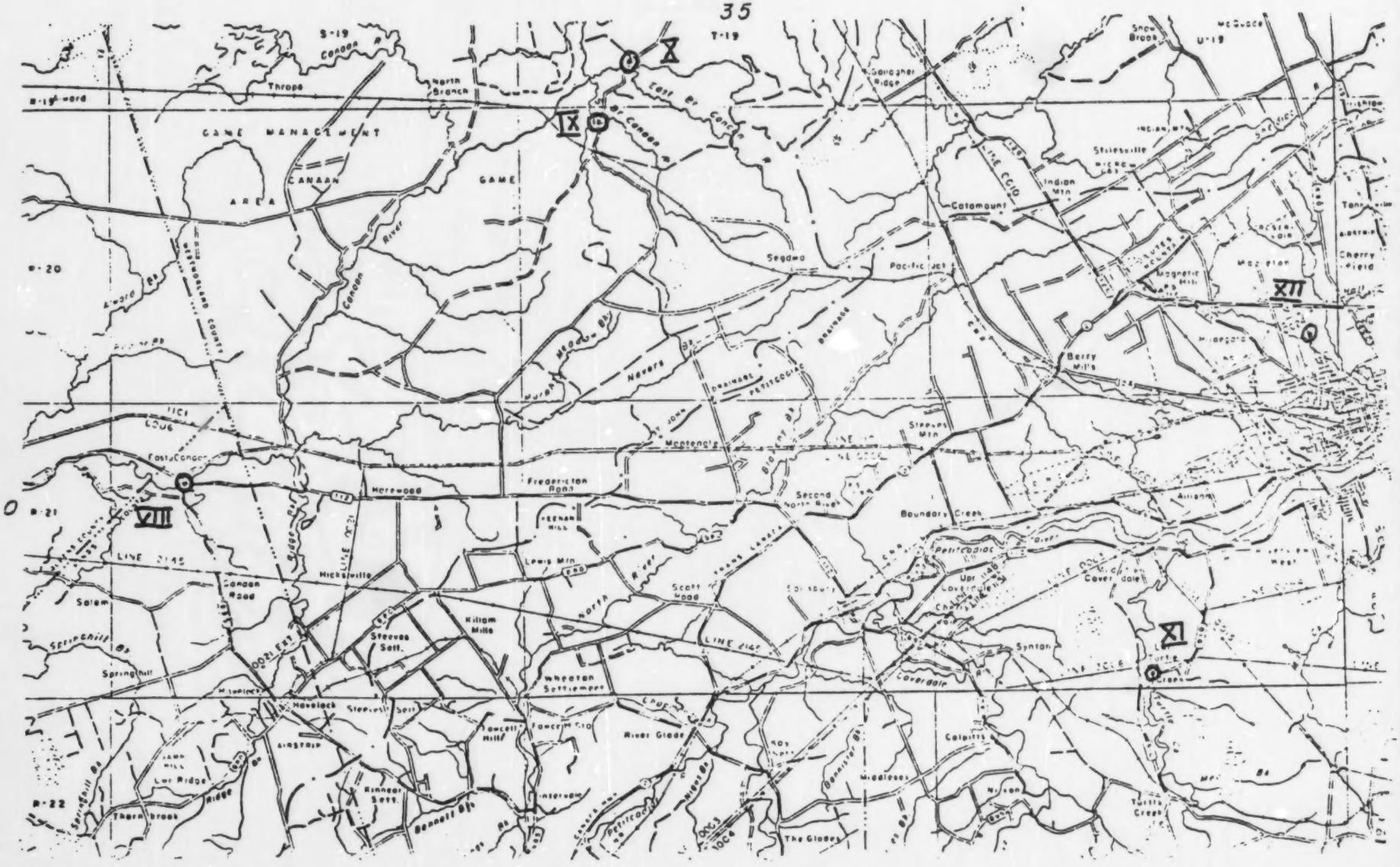

40 cm deep) and at least four metres from the shore at all sites except in brooks where sampling was done in the middle of the stream. With the exception of Site VII (beaver dam) the water was fast-flowing. Sampling was done with a 2-liter long-necked plastic bottle (lined with lead strips for weight).

Spray information regarding blocks sprayed near sampling areas is given in Table 1 (refer to Figure 2).

MATERIALS AND METHODS

Chemicals and Apparatus

(1) All the organic solvents used were "pesticide grade". A stock solution of fenitrothion (Forest Protection Ltd.) was prepared 1 µg/µl (1000 ppm) from which dilutions in ethyl acetate were made for gas liquid chromatographic (glc) calibration.

(2) Amberlite resin XAD-4 was obtained from BDH CHEMICALS (Halifax, N.S.). Purified Amberlite XAD-2 resin was obtained from TETROCHEM LAB (Edmonton, Alberta).

(3) A gas liquid chromatograph (Perkin-Elmer 3920) fitted with a flame photometric detector (FPD) and a glass column (6 mm I.D. x 1.8 m containing 3.6% OV-101 and 5% OV-210 on CHROMOSORB W, DMCS 80/100, was used for all quantitative analyses.

(4) Portable air pumps (no. 1944) used for air sampling were purchased from Lamotte Chemical (Chesterton, MA, U.S.A.). For prolonged air sampling a GELMAN Inst., MODEL 13152 pressure/vacuum pump was used. Glass columns 2.1 cm I.D. x 29 cm long with an elongated tip (I.D. 5 cm x 1 cm) were

Table I

Spray Information Per Area

(Refer to pages 7 and 12)

Area I

Block no.	Spray dates
(27)	June 4, 7; June 15, 17
(28)	June 4, 5, 6; June 15, 17, 19
(29)	June 3, 5, 6, 7; June 15, 16, 17
30	June 3, 4, 5, 6, 7; June 13, 15, 16, 17
31	June 3, 4, 5; June 10, 11, 12, 13
(37)	May 29; June 4
39	May 31; June 4, 6, 10, 11, 12
40	May 31; June 2, 4; June 12, 13
(41)	June 1, 2, 4; June 11, 12
42	June 3, 4; June 13

Area II

Block no.	Spray dates
50	May 28, 29; June 2, 3

Area III

(not sprayed)

Area IV

(not sprayed)

N.B. Numbers without brackets indicate sampling site at/or near sprayed blocks.

Numbers with brackets indicate sampling sites nearby.

prepared in our laboratory from ordinary glass tubing.

Experimental Procedures

(1) Preparation of XAD columns

The procedures for the preparation, cleaning and regeneration of the columns for water extraction have already been described (6). For air sampling, a glass column was filled with the resin to a height of 12 cm and used as such.

(2) Treatment of water samples

A one liter sample was passed through a column on-site by gravity flow. The columns used in Area 1 were kept in the field for one week prior to transportation to the lab where they were usually processed the following week.

(3) Treatment of air samples

In Area I, sites I and II, air was drawn through a column at a rate of 2L per min for a total of 100L. At Area IV, site I, air was drawn through the column at a rate of 2L/min for 24h.

(4) Processing of column and quantitation

Each column was eluted with 3 x 30 mL of ethyl acetate. The eluate was concentrated to 10 mL and a 5 µl aliquot was injected into the gas chromatograph. Standards were also injected for calibration purposes. Each column was regenerated by washing successively with 50 mL of acetone,

50 mL of methanol and 100 mL of distilled water for further utilization. The oven temperature of the chromatograph was 190°C. Periodic (every 50 samples) reagent blank checks were made to verify for possible contamination of apparatus. None was ever found.

(5) Confirmation of identity

The procedure for the detection of fenitrothion and aminofenitrothion by gas chromatography is already well established in our laboratory (6). Confirmation of the presence of fenitrothion and aminofenitrothion was done using a different instrument (Tracor 560 equipped with a nitrogen-phosphorous detector) and a different column (OV-17).

Spray Regime

In all the areas surveyed for the presence of fenitrothion the spray regime consisted of two applications of fenitrothion (active ingredient) at 210g/ha. The formulation contained by weight 14.5% technical fenitrothion, 1.4% Dowanol (a solvent), 1.5% Atlox 3409F (emulsifier), and 82.6% water.

RESULTS

The data from samples of the Canaan River area are summarized in Table II. At the East Branch site, fenitrothion was detected

the day after the first application on spray block 50 (according to the dates supplied by FPL, ANNEX 2 and Table I). This can be explained because sampling was done in the morning while the first application actually occurred in the afternoon of May 28. A maximum of 2.8 ppb was observed May 29 but no trace of fenitrothion was found May 31. Fenitrothion was again detected on June 3 corresponding with the second application on June 2 (p.m.) but again nothing was detectable a few days later.

In the South Branch the phenomenon was very similar except that traces of fenitrothion were observed on the same day as spraying started. It is not certain whether sampling was actually carried out before or after the first application since the exact time of spraying was not known to us. However, the information should be available from FPL upon request. A maximum of 1.7 ppb was detected and post-spray sampling indicated no persistence of fenitrothion.

In the main body, i.e., the Canaan River at East Canaan, the data are similar to those obtained from the two feeding brooks. Thus a maximum of 3.5 ppb was detected and the appearance of fenitrothion seemed to correspond with the first application. Persistence was also not a problem. There were duplicate samples picked up June 5, one of which was extracted using solvent-solvent partition (chloroform) and the other, an XAD-4 column. The results are comparable (also see data for East Branch) indicating that the use of an XAD-4 column is

reliable. This reliability has already been demonstrated in the literature (6).

Fenitrothion was never detected in samples coming from the Turtle Creek Reservoir (Table III) over a 30-day period. However spraying was not carried out in close proximity to the reservoir except perhaps at the end of June when blocks 48 and 49 were sprayed. Nonetheless since contamination never occurred, transportation via spray drifts seems to have been insignificant.

In the Nipisiguit River Area, three sites were established as follows: at Forty Four, across Route 430, and at Bathurst Mines Dam. At Forty Four (Table IV), daily samples taken starting June 1 revealed traces of fenitrothion intermitently throughout the sampling period. The amounts were usually very low (except possibly on June 20 when 20.0 ppb were detected), but the appearance and disappearance of fenitrothion cannot really be directly related to a particular spray date or block although it may be said that spraying was occurring in the general area of the site at the time. Fenitrothion was detected June 27 and this cannot be explained on the basis of date versus block because spraying was completely done by June 20. This does not exclude the possibility of contamination because of rain washouts or drifts from other areas (possibly Quebec) but this latter assertion would have to be verified to determine whether or not Quebec was spraying at the time. Samples from

July 5 and 9 failed to reveal any trace of fenitrothion indicating no persistence.

The detection of high concentrations of aminofenitrothion relative to fenitrothion on certain days is puzzling. Aminofenitrothion is a product of reductive anaerobic degradation and should not usually be detected in free flowing water that contains oxygen. It is also interesting to note that aminofenitrothion was detected in samples that did not show any trace of fenitrothion. The possibility that the reduction may have occurred in the column seems unlikely since laboratory tests have shown that fenitrothion remains stable for a prolonged time in an XAD column. However, the degradation of fenitrothion in XAD columns with time under field conditions has never been studied although environmental samples have been shown to be stabilized by the resin (6).

In the Nipisiguit River across Route 430, sampling was started May 27 and done twice a day, morning and afternoon, to obtain more representative information. The data presented in Table V show that traces of fenitrothion first appeared June 5 (as was the case at Forty Four), and intermitently from then on. A maximum of 14.0 ppb was observed on June 15 and the concentration remained at that level for a few more days. On the contrary, data at Forty Four (Table IV) were more erratic and amounts were less concentrated. The disappearance of fenitrothion seems to correspond approximately with the end of spraying (June 19)

in that area. Traces of aminofenitrothion were observed on occasion.

At Bathurst Mines Dam (Table VI) traces of fenitrothion were observed earlier on June 3. Otherwise the data are comparable to those obtained at Forty Four including the presence of fenitrothion June 27. Traces of aminofenitrothion were also observed and the disappearance of fenitrothion seems to correspond with the end of spraying.

Two brooks, namely Big Elbow and at Forty Four, feeding into the Nipisiguit River, were also investigated starting May 27. The data at Big Elbow (Table VII) show the presence of fenitrothion June 4, and for four consecutive days thereafter. Appearance is again noted June 15 with a maximum of 13.8 ppb June 18. Fenitrothion was not detected after June 23 which corresponds with termination of the spray program. At Forty Four (Table VIII) fenitrothion was detected as early as May 27 which is difficult to relate to spraying in the area. In addition fenitrothion and aminofenitrothion were observed more often than at Big Elbow. A maximum of 9.7 ppb of fenitrothion was noted June 20 following seven consecutive days of detection. Aminofenitrothion was detected on June 26 which is difficult to interpret.

The only other site for water sampling was a small beaver pond along Route 430 not very far from Sevogle airstrip somewhere

inside block 40 (see Figure 2). Sampling was done twice a day morning and afternoon starting May 27. Fenitrothion was first detected June 1 (Table IX) and its presence (and sometimes that of aminofenitrothion) was observed daily until June 20 (except for June 19). Actually block 40 was first sprayed May 31 which explains the appearance of fenitrothion June 1. In fact spraying was done in the immediate vicinity of block 40 almost everyday until June 13 (see Table I and Figure 2). A maximum of 11.8 ppb was detected June 15 corresponding with higher levels around that date. Nothing was detected after June 23.

Air sampling was carried out at three sites, two of which were in the Sevogle Area, the other on campus at Université de Moncton, to act as control. The amounts detected at Sevogle airstrip (Table X) were very low (traces indicate less than 0.5 ng per dm^3 of air. More interesting, however, was the presence of aminofenitrothion which was detected quite often, and with a maximum concentration of 12.0 ng/dm^3 on June 17. At the Nipisiguit River site across Route 430 air sampling was also done twice a day. Fenitrothion was detected above detection limit only a few times but aminofenitrothion was also present more often than the parent compound although the quantities detected were low (Table XI). While traces of AF were detected as late as June 24 at Sevogle airstrip nothing

was observed at the Nipisiguit River site after June 19. At the third site (in Moncton) sampling was done on a 24h basis thus lowering the limit of detection by a factor of 25. Fenitrothion was detected on June 4 and 5 only (Table XII).

DISCUSSION

The data obtained from the Canaan River area suggest that the contamination of small brooks feeding into a main body in an isolated small block, as a result of spraying operations such as those carried out this year, is very light and short-lasting. Also the results suggest that contamination of the main body by the feeding brooks is not serious and lasting.

The data from the Turtle Creek Reservoir showed no contamination over the entire spray period which seems to indicate that contamination of such a water body as a result of drift or other entry is not important. However, contamination by drift is probably highly dependent on wind conditions and precipitation and no definite conclusions can be drawn since only one site was sampled.

In the Nipisiguit River Area spraying was done over a very large surface for over three weeks. It is thus difficult to relate the results to any particular spray date or block because entry at the sampling sites was possible from many directions. However, since most of the concentrations were

below 10 ppb, except in a few instances, it may be suggested that contamination of a river such as the Nipisiguit (in size, volume, and movement) is light and short lasting. The appearance of aminofenitrothion, however, is difficult to interpret as was mentioned earlier (see RESULT). At times AF only was detected (no Fenitrothion). At the Route 430 site on the Nipisiguit River sampling was done twice a day. The magnitude of the results from samples picked in the afternoon was usually similar to that of the results from the morning suggesting that a good estimate of the contamination could have be obtained from one sample per day, thus saving in energy and cost. Results obtained from the brooks feeding into the Nipisiguit River also showed light contamination which was short-lasting.

The data from Sevogle Pond (beaver pond on Route 430 close to Sevogle airstrip) seem to indicate that contamination in a still body of water is more persistent. However, it is difficult to make more general conclusions because spraying in the close vicinity of the site (blocks 37, 39, 40, 41) occurred over a period of several days. It should be noted that although samples picked the same day yielded comparable data, results from the morning were usually higher than those from the afternoon. It is presumed that greater degradation or loss by evaporation occurred during the day than at night accounting for lower concentrations in the afternoon.

The amounts of fenitrothion detected in air anywhere were always extremely low. Amazingly AF was detected sometimes in

concentrations greater than 10 ng per dm^3 while fenitrothion was not present. As mentioned earlier the aminofenitrothion phenomenon remains unclear and more work will have to be done to determine its origin. The data at Sevogle airstrip, which was an area completely surrounded by spray zones, suggests that air contamination as a result of drift at ground level is very light not to say insignificant. Evidently one would expect to find higher levels if sampling was done under a spray cloud but that was not the intent here. Similar reasoning is supported by data at the Nipisiguit River air sampling site. The data from Moncton, a typical non-sprayed urban area, suggest that light contamination is possible probably being highly dependent on weather conditions. To be more confident in making conclusions, more sites would have to be established at strategic places in the Province. It seems also very important that 24h air sampling be carried out such that total accumulation could be documented. Choosing a particular hour to sample is not appropriate because air contamination is likely to vary from nil to high depending on the time of day.

CONCLUSIONS

The most important fact that emerges from this study is that the contamination of water and air by fenitrothion in the areas surveyed was very light. The total number of

samples analyzed was 465 far more than any other survey of a similar nature carried out in New Brunswick in previous years. The number of samples treated is considered sufficient to draw tentative conclusions with regards to the particular sites studied. It does not permit, however, to draw general conclusions for the entire Province since only ten sites were sampled for water and only three sites were selected for air sampling. Nonetheless the information gathered this summer will be valuable over a period of years (let's say five years) since it is expected that eventually a pattern of contamination will emerge from subsequent studies.

One important conclusion can be drawn in relation to the number of samples necessary per day. At the sites where two samples were picked each day the data from the second sampling usually confirmed the first. So in evaluating the level of contamination in water it is felt that sampling once a day should be sufficient. It is reminded that the object of this study was not to look at variations in concentrations within a day but rather variations in concentrations during the entire spray programme. For air it is different, contamination is likely to vary from hour to hour. 24h monitoring is preferable.

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RECOMMENDATIONS (in relation with the annual spray programme to control spruce budworm)

(1) that a chemical residue survey be repeated in 1981 and as long as required.

(2) that the analysis include the degradation products and the parent compounds used as insecticides.

(3) that the analysis include the additives in the formulations, particularly with air samples.

(4) that the substrates include water, air, foliage, soil litter and biota samples.

(5) that the sampling sites be located at different places every year such that eventually the results may be considered representative of the Province as a whole

or

that the sites remain fixed but be distributed uniformly throughout the Province so as to ensure representation.

(6) that a Committee be set up to coordinate the exercise in such a way that a minimum number of samples be processed to obtain maximum valuable information.

Table II

Fenitrothion (ppb) in Water From Canaan River Area

(Block 50/250)

Sampling date	East Branch Canaan River	South Branch Canaan River	Canaan River at East Canaan
May 14, 1980	N.D.	N.D.	N.D.
27	N.D.	N.D.	N.D.
28	N.D.	Traces	0.5
29	2.8	0.6	N.D.
30	0.7	0.9	3.5
31	N.D.	Traces	0.5
June 1, 1980	N.D.	Traces	Traces
2	N.D.	N.D.	N.D.
3	0.5	Traces	0.8
4	3.4	1.7	2.6
5	0.9	0.8	Traces
5[a]	0.7	-	Traces
19	N.D.	N.D.	N.D.

LEGEND:

N.D.: means not detectable
(-) : means no sample was taken
Traces: the quantitative detection limit is 0.5 ppb. Traces indicate quantities between 0.05 - 0.5 ppb.
a: indicates a duplicate sample extracted with chloroform.

Total no. of samples: 38

Table III

Fenitrothion (ppb) in Water From Turtle Creek

Sampling date	Concentration	Sampling date	Concentration
May 14, 1980	N.D.	June 10, 1980	N.D.
27	N.D.	11	N.D.
28	N.D.	12	N.D.
29	N.D.	13	N.D.
30	N.D.	14	N.D.
31	N.D.	15	N.D.
		16	N.D.
June 1, 1980	N.D.	17	N.D.
2	N.D.	18	N.D.
3	N.D.	19	N.D.
4	N.D.	20	N.D.
5	N.D.	21	N.D.
6	N.D.	22	N.D.
7	N.D.	23	N.D.
8	N.D.	24	N.D.
9	N.D.		

Total no. of samples: 30

Table IV

Fenitrothion (ppb) in Water From The Nipisiguit River at Forty Four

Sampling date	Forty Four	Sampling date	Forty Four
May 21, 1980	N.D.	June 13, 1980	N.D. (8.0 A.F.)
27	-	14	N.D. (2.8 A.F.)
28	-	15	5.3
29	-	16	1.3
30	-	17	N.D.
31	-	18	N.D.
		19	3.2
June 1, 1980	N.D.	20	20.0
2	N.D.	21	-
3	N.D.	22	-
4	N.D.	23	N.D.
5	0.9	24	N.D.
6	1.0	25	N.D.
7	N.D.	26	N.D.
8	N.D.	27	1.6 (3.0 A.F.)
9	N.D. (A.F.)		
10	N.D.	July 5, 1980	-
11	N.D.	9	N.D., N.D.
12	N.D.		

LEGEND:

(A.F.): means aminofenitrothion indicated to the right next to fenitrothion. Amount greater than 0.5 ppb are indicated.

Total no. of samples: 28

Table V

Fenitrothion (ppb) in Water From The Nipisiguit River at Bridge on Route 430

Sampling date	Bridge on Route 430	
	A.M.	P.M.
May 21, 1980	-	N.D.
27	N.D.	-
28	N.D.	-
29	N.D.	-
30	N.D.	-
31	N.D.	-
June 1, 1980	N.D.	N.D.
2	N.D.	N.D.
3	N.D.	N.D.
4	N.D.	N.D.
5	1.9	1.3
6	N.D.	0.8
7	Traces	Traces
8	Traces	Traces
9	Traces (A.F.)	N.D.
10	N.D.	N.D.
11	N.D.	1.0 (A.F.)
12	N.D.	N.D.
June 13, 1980	Traces(A.F.)	2.8
14	N.D.	N.D.(A.F.
15	11.0	14.0
16	10.5	12.9
17	10.0	11.2
18	13.2	9.3
19	9.1	8.2
20	8.0	8.8
21	-	-
22	-	-
23	N.D.	N.D.
24	N.D.	N.D.
25	N.D.	N.D.
26	N.D.	N.D.
27	N.D.	N.D.
July 5, 1980	-	N.D.
9	N.D.,N.D.	

Total no. of samples: 59

Table VI

Fenitrothion (ppb) in Water From The Nipisiguit River at Bathurst Mines Dam

Sampling date	Baturst Mines Dam	Sampling date	Bathurst Mines Dam
May 21, 1980	N.D.	June 13, 1980	N.D. (2.5 A.F.)
27	N.D.	14	1.8
28	-	15	4.1
29	-	16	N.D.
30	-	17	Traces
31	-	18	8.3
		19	12.9 (3.1 A.F.)
June 1, 1980	-	20	N.D.
2	N.D.	21	-
3	Traces	22	-
4	0.5	23	N.D.
5	2.1	24	N.D.
6	0.8	25	N.D.
7	N.D.	26	N.D.
8	N.D.	27	2.31
9	N.D.		
10	N.D. (3.2 A.F.)	July 5, 1980	N.D.
11	N.D. (A.F.)	9	N.D., N.D.
12	N.D. (A.F.)		

Total no. of samples: 29

Table VII

Fenitrothion (ppb) in Brooks Feeding Into The Nipisiguit River at Big Elbow Brook

Sampling date	Big Elbow Brook	Sampling date	Big Elbow Brook
May 21, 1980	N.D.	June 13, 1980	N.D.
27	N.D.	14	N.D.
28	N.D.	15	1.2
29	N.D.	16	12.5
30	N.D.	17	N.D.
31	N.D.	18	13.8
		19	11.0(A.F.)
June 1, 1980	N.D.	20	7.1
2	N.D.	21	-
3	N.D.	22	-
4	1.6	23	N.D.
5	0.9	24	N.D.
6	0.8	25	N.D.
7	Traces	26	N.D.
8	Traces	27	N.D.
9	N.D.		
10	N.D.	July 5, 1980	-
11	N.D.	9	N.D., N.D.
12	N.D.		

Total no. of samples: 33

Table VIII

Fenitrothion (ppb) in Brooks Feeding Into The Nipisiguit River at Forty Four

Sampling date	Forty Four	Sampling date	Forty Four
May 21, 1980	N.D.	June 13, 1980	N.D.
27	4.4	14	N.D. (A.F.)
28	-	15	9.1
29	-	16	1.6
30	-	17	1.4 (A.F.)
31	-	18	8.0 (7.3 A.F.)
		19	8.0
June 1, 1980	-	20	9.7
2	2.0	21	-
3	N.D.	22	-
4	N.D.	23	N.D.
5	0.9	24	N.D.
6	1.7 (3.5 A.F.)	25	N.D.
7	Traces	26	N.D. (2.0 A.F.)
8	Traces	27	N.D.
9	N.D.		
10	N.D.	July 5, 1980	N.D.
11	N.D.	9	N.D., N.D.
12	N.D. (A.F.)		

Total no. of samples: 29

Table IX

Fenitrothion (ppb) in Sevogle Pond

Sampling date	A.M.	P.M.	Sampling date	A.M.	P.M.
May 21, 1980	N.D.	N.D.	June 14, 1980	0.9	0.6 (A.F.)
27	N.D.	N.D.	15	11.8	4.6
28	N.D.	N.D.	16	11.4	Traces
29	N.D.	N.D.	17	8.9	6.0
30	N.D.	N.D.	18	4.1	Traces (A.F.)
31	N.D.	N.D.	19	N.D.	N.D.
			20	6.4	4.3
June 1, 1980	3.2	2.8	21	-	-
2	1.9	2.3	22	-	-
3	2.2	1.9	23	N.D.	N.D.
4	1.5	0.9	24	N.D.	N.D.
5	0.7	0.6	25	N.D.	N.D.
6	0.8	0.5	26	N.D.	N.D.
7	Traces	Traces	27	N.D.	N.D.
8	0.5	2.0			
9	2.2	N.D.	July 5, 1980	N.D.	-
10	Traces	Traces	9	N.D.,	N.D.
11	2.6	0.9			
12	1.7 (A.F.)	1.1			
13	0.6 (A.F.)	Traces (A.F.)			

Total no. of samples: 67

Table X

Fenitrothion (ng/dm^3) in Air From Sevogle Area at Sevogle Airstrip

Sampling date	Sevogle Airstrip A.M.	Sevogle Airstrip P.M.	Sampling date	Sevogle Airstrip A.M.	Sevogle Airstrip P.M.
May 21, 1980	N.D.	N.D.	June 13, 1980	Traces	N.D.
27	N.D.	N.D.	14	N.D.	Traces (A.F.)
28	N.D.	N.D.	15	N.D.	N.D. (1.3 A.F.)
29	N.D.	N.D.	16	N.D.	N.D.
30	N.D.	N.D.	17	N.D.	N.D. (12.0 A.F.)
31	Traces	N.D.	18	N.D.	N.D.
June 1, 1980	N.D.	N.D.	19	N.D. (4.6 A.F.)	N.D.
2	N.D.	N.D.	20	N.D.	N.D.
3	N.D.	N.D.	21	-	-
4	N.D.	N.D.	22	-	-
5	N.D.	Traces	23	N.D.	N.D.
6	N.D.	N.D.	24	N.D.	N.D. (2.0 A.F.)
7	N.D.	N.D.	25	N.D.	N.D.
8	N.D.	N.D.	26	N.D.	N.D.
9	N.D.	N.D.	27	N.D.	N.D.
10	N.D.	N.D.			
11	Traces	Traces (A.F.)			
12	N.D.	N.D.			

LEGEND:

The quantitative detection limit is 0.5 ng/dm^3.

Traces indicates quantities between 0.05-0.5 ng/dm^3.

Total volume: 100L per sample

Total no. of samples: 62

Fenitrothion (ng/dm^3) in Air From Sevogle Area at Nipisiguit River at Bridge

Sampling date	Nipisiguit River at Bridge		Sampling date	Nipisiguit River at Bridge	
	A.M.	P.M.		A.M.	P.M.
May 21, 1980	N.D.	N.D.	June 13, 1980	N.D.	N.D.
27	N.D.	-	14	N.D.	N.D.
28	N.D.	-	15	N.D.	N.D.
29	2.0	-	16	N.D.	N.D.
30	N.D.	-		(A.F.)	(5.7 A.F.)
31	N.D.	-	17	N.D.	N.D.
				(6.4 A.F.)	(5.0 A.F.)
June 1, 1980	N.D.	N.D.	18	N.D.	N.D.
2	N.D.	N.D.	19	N.D.	N.D.
3	N.D.	N.D.	20	N.D.	N.D.
4	N.D.	N.D.	21	-	-
5	N.D.	N.D.	22	-	-
6	N.D.	N.D.	23	N.D.	N.D.
7	0.7	N.D.	24	N.D.	N.D.
8	N.D.	N.D.	25	N.D.	N.D.
9	N.D.	N.D.	26	N.D.	N.D.
10	N.D.	N.D.	27	N.D.	N.D.
11	N.D.	N.D.			
12	N.D.	N.D.			

Total no. of samples: 57

Table XII

Fenitrothion in Air From Moncton (on Campus at U. de Moncton)

Sampling date	Concentration (ng/dm^3)
May 23 - June 3rd	N.D.
June 4, 1980	0.07
5	1.2
June 6 - June 23rd	N.D.

LEGEND:

The quantitative detection limit is 0.02 ng/dm^3

Total volume: 2880L per sample

Total no. of samples: 33

ANNEX 1

SUMMARY OF WATER QUALITY BRANCH PESTICIDE STUDIES

1976-1980

WATER QUALITY BRANCH

INLAND WATERS DIRECTORATE

ENVIRONMENT CANADA

ATLANTIC REGION

Fenitrothion in Soil Litter (1976)

A short survey to determine the concentration and persistence of fenitrothion and its degradation products in upper soil horizons was conducted during the summer of 1976. Soil samples were collected from three sprayed blocks during three distinct sampling periods; before aerial spraying, shortly after aerial spraying and one to two months after aerial spraying.

Fenitrothion was only occasionally detected in the soil samples and only in those collected immediately after aerial spraying. On the other hand, most soil samples revealed the continued presence of DDT, its isomers and degradation products, presumably from aerial spraying operations conducted in the 1960s.

Fenitrothion in Water and Sediments from Streams, Bogs and Swamps (1976)

During the summer of 1976, the Water Quality Branch conducted a study designed to determine the concentration and persistence of fenitrothion and its degradation products in water and sediments from streams, bogs and swamps. Samples of each substrate were collected from three sprayed blocks during three distinct samplings;before aerial spraying, shortly after aerial spraying and one to two months after aerial spraying. In the second sampling period, water samples were collected at intervals of 1, 2, 4, 8, 12 and 24 hours after aerial spraying.

Fenitrothion was found in about half of the eighty samples collected directly after application of the spray, but was not detected in any samples taken before spraying or in samples collected one to two months after aerial application. Shortly after spraying, extremely high concentrations of fenitrothion were found in a surface water from the swamp area. Concentrations of fenitrothion in other stationary waters and in flowing waters ranged from moderate to barely a trace. In surface waters, maximum concentrations were reached two to four hours after aerial spraying and decreased rapidly thereafter to the point of being extremely low or non-detectable after 24 hours. The decrease of fenitrothion with time was not coupled with an increase in its major degradation products. FenitroOxon, aminofenitrothion and nitrocresol were rarely detected and when detected were found to be in very low concentrations.

Ground Truthing for Fenitrothion (1977)

One study conducted by the Water Quality Branch in 1977 was to assess the uniformity of the fenitrothion application to a 500 m rectangular grid. Glass petri dishes containing XAD-2 resin were placed on a measured 500 metre grid in an open field shortly before aerial spraying and were collected two hours after spraying. Analysis of the samples indicated a seven-fold difference in local deposition across this flat open area terrain. The average accountancy was only 28% of the reported application.

Suspended Sediment Study (1977)

The objective of this study was to determine if fenitrothion and aminocarb adhered to suspended sediments in flowing waters, and if so, to what extent could this mechanism account for the overall transport of these pesticides through the aquatic environment.

Suspended sediments were collected from the Cains and Upsalquitch Rivers by passing 600 litres of river water through a continuous flow centrifuge. Sampling was conducted 1, 2, 4, 8, 12, 24, 48 and 72 hours after aerial spraying. The amount of suspended sediments collected varied from six grams directly after a storm to less than one gram under normal conditions. The results indicated that the amount of pesticide in the suspended sediments varied from 0.02 to 1.2 mg/Kg during the three days of surveillance with the higher concentrations being detected shortly after spraying. In comparing the concentration of insecticide in the suspended sediment to that in the water, the results show that less than 1% is associated with the suspended sediments.

Fenitrothion in a Still Water Pond (1977)*

Water, suspended solids and sediment samples were collected from a small pond before and after aerial spraying of fenitrothion and the samples were analyzed for fenitrothion and its degradation and transformation products. Fenitrothion concentrations in water, suspended

solids and sediment fell below detectable levels two days after the spray. The only identified products were p-nitro-m-cresol in water, which persisted less than two days, and aminofenitrothion in sediment, which persisted less than four days. Laboratory experiments indicated that chemical hydrolysis and volatilization of fenitrothion from true solution were both slow processes; however, volatilization of fenitrothion from surface slicks of its formulation sprayed in water was very fast.

* Major work done by CCIW and supported by WQB in the Atlantic Region. Paper published by Maguire and Hale, J. Agric. Food Chem 28, 372 (1980).

A Comparative Study of the Use of XAD-2 Resin and the Conventional Serial Solvent Extraction Procedure for the Analysis of Fenitrothion and Some Derivatives in Water Preservation Techniques (1978)*

An analytical technique using the macroreticular resin XAD-2 for recovering fenitrothion, fenitroOxon and aminofenitrothion in water was studied. The procedure was evaluated directly in the field immediately after an aerial application of the formulated insecticide. Results compared well with those obtained by a modified serial solvent extraction method. It was also found that water samples containing fenitrothion and the two derivatives could be processed on XAD-2 columns and that the chemicals would be stabilized for at least six days.

* Mallet, Brun, MacDonald and Berkane, J. Chromatog. 160, 81 (1978).

High Performance Liquid Chromatography Analysis of Aminocarb; Ultraviolet Absorbance and Fluorescence Detection in Series (1978)

A sensitive analytical procedure, amenable to routine analysis of aminocarb at residue levels was developed. The basic system consisted of a high performance liquid chromatograph equipped with a reverse-phase column, UV-absorbance and fluorescence detector in series.

The separation of mixed components was accomplished by isocratic elution with a mobile phase consisting of a phosphate buffer and acetonitrile. Minimum detectable amounts for aminocarb in water was 0.05 ug/L.

One major advantage of using this system was that the separation was carried out at ambient temperature thus overcoming problems associated with the decomposition of carbamates and the elimination of the need to prepare derivatives required by GLC analysis. The method was also found applicable to sediment, soil and foliage without prior column clean-up.

Analysis of Fenitrothion in Rain Water using an XAD-4 Resin Column (1978)

An analytical method was adopted for the analysis of fenitrothion in rain water. The method consist of passing the rain water through an XAD-4 column and then eluting the column, with ethyl acetate in order to extract the fenitrothion. The ethyl acetate solution is then dried and concentrated. The concentrate is analyzed for the insecticide by gas liquid chromatography.

Off-Target Fallout of Fenitrothion During 1978 Forest Srpaying Operations in New Brunswick (1978)*

A study was conducted to determine if the sprayed insecticide was carried in the prevailing winds and deposited in far field locations either as dry fall-out or as rain wash-out. Collectors (funnel attached to an XAD-4 resin column draining into an amber glass bottle) were deployed at 12 sites in eastern N. B. immediately prior to the insecticide spraying operation in 1978. All sites (11 outside spray zones and one inside a spray zone) were visited on a weekly basis and the resin columns were eluted with ethyl acetate. These samples were subsequently analyzed in the laboratory for fenitrothion using glc.

Outside the spray zones, fenitrothion was found in one-half of the samples and ranged from 0.01 ug/L to 1.1 ug/L. In the spray zone the fenitrothion was found to be as high as 77 ug/L. It was concluded that off-target fallout of fenitrothion was minimal during the 1978 spray operation.

* Pearce, Brun & Witteman; Bull Environm. Contam. Toxicol. 23, 504 (1979).

Persistence and Vertical Distribution of Fenitrothion in a Lake (1978)

The aim of this preliminary study was to determine if there existed a temporal and vertical distribution of fenitrothion in lake water following aerial spraying. The experimental site was Peabody Lake which was situated in a fenitrothion spray zone. Water samples were collected

over a 24 hour period at 1 meter below the surface of the lake (surface sample) and 3 meters below the surface (bottom sample). The fenitrothion concentration in the surface samples ranged from 0.62 ug/L one hour after spraying to 1.34 ug/L 24 hours after spraying. On the other hand, bottom samples ranged from 0.02 ug/L one hour after spraying to 1.13 ug/L 24 hours after spraying. These results confirmed temporal variation and vertical distribution in the first 24 hours.

Persistence and Vertical Distribution of Aminocarb in a Lake (1978)

The aim of this preliminary study was to determine if there existed a temporal and vertical distribution of aminocarb in lake water following aerial spraying. The experimental site was Nictau Lake which was situated in an aminocarb spray zone. Water samples were collected over a 48 hour period at 1 meter below the surface of the lake (surface sample) and 10 meters below the surface (bottom sample). Aminocarb was not detected in any of the samples, therefore making it impossible to draw any concrete conclusions.

Tobique River Study (1978)

The Tobique River which drains Nictau Lake was sampled over a 36 hour period at Riley Brook in order to gather data on the persistence of aminocarb in flowing water. This study was conducted concurrently with the previously mentioned investigation. This sampling site was situated 52 Km below Nictau Lake and 10 Km outside the spray zone. Aminocarb was not detected in any of the seven samples collected again making it impossible to draw any definite conclusions.

Use of Freshwater Mussels as Indicators of Fenitrothion Contaminated Waters (1978)

A feasibility study was conducted to determine the possibility of employing caged freshwater mussels as indicators of fenitrothion contamination in a small lake. The site selected for this investigation was Peabody Lake in Northeastern New Brunswick. Freshwater mussels were collected from the lake prior to the spray operation and placed in cages which were anchored to the bottom of the lake and given time to acelimatize. The intent was to remove two mussels per day from each cage for ten days commencing one day before spraying. All mussels were to be shucked, frozen and returned to the laboratory for fenitrothion analysis.

The investigation was plagued with problems including equipment failures, indicator mortality and vandalism. Although some mussels were finally returned to the laboratory for analysis and that all results were negative, definite conclusions could not be drawn.

Sampling of Estuarine and Lake Sediments for Insecticide Residues (1978)

The purpose of this study was to determine if bottom sediments are acting as a sink for fenitrothion reaching the water column. Sediment samples were collected from the Miramichi estuary, Peabody Lake and Lake Nictau and returned to the laboratory for fenitrothion analysis.

Analyses indicated no parent pesticide in any of the sediment samples.

Determination of Nonyl Phenol in Water and Sediment by High Performance Liquid Chromatography (1979)

A method was developed for the analysis of nonyl phenol at levels in both water and sediment. The procedure consist of serial solvent extraction followed by high performance liquid chromatography. The liquid chromatography separation was performed isocratically on a reverse-phase column using a mobile phase system composed of acetonitrile and water and detection was accomplished using a UV detector. Detection limits of 1.0 ug/L for water and 0.3 mg/Kg for sediments were achieved.

Off-Target Fallout of Aminocarb During 1979 Forest Spraying Operations in New Brunswick (1979)

A study was conducted to determine if the sprayed insecticide was carried in the prevailing winds and deposited in far field locations either as dry fall-out or as rain wash-out. Collectors (funnel attached

to an XAD-4 resin column draining into an amber glass bottle) were deployed at 9 sites in eastern N. B. immediately prior to the insecticide spraying operation in 1979. All sites were visited on a bi-weekly basis and the resin columns were eluted and the extracts were subsequently analyzed for aminocarb by HPLC.

It was observed that one-third of the samples contained aminocarb. Aminocarb was found in all but one sample collected during the first two weeks of June, when 60% of the insecticide used in the spray operation was applied. The average concentration in the positive samples from outside the spray blocks was 0.15 ug/L. This study illustrated that forest insecticides could be transported by the atmosphere considerable distances from the target.

Study on the Persistence of Nonyl Phenol in an Aquatic System (1979)

The aim of this study was to determine the persistence of nonyl phenol in the water and sediment compartments of a small lake. The lake selected for this study was Black Lake located in southeastern New Brunswick.

Water and sediment samples were collected both before and after aerial spraying but the results showed no detectable amounts of nonyl phenol in any of the samples.

Off-Target Fallout of Fenitrothion (1980)

The aim of this 1980 pesticide study was to determine, in non-target areas, the fraction of aminocarb deposited as dry fallout and that deposited as rain washout. Bulk and event collectors were placed in two non-target areas (Fundy National Park and CFB Saint Mary). The bulk collectors were constantly open to the atmosphere while the event collectors were only opened when it rained. These collectors were installed one week before the 1980 aerial spraying operation and remained in place until one week after the spraying operation. Samples were returned to the laboratory for fenitrothion analysis on a weekly basis. The data is presently being analyzed.

Dr. P.E. Belliveau
August, 1980

ANNEX 2

1980 FENITROTHION BLOCKS VERSUS SPRAY DATE

May 24 A.M. - 71, 70, 69, 77, 78, 79, 80

P.M. - 68, 69, 67, 63, 58

May 25 A.M. - 56, 64, 61, 57, 85, 82, 83, 80, 79

May 26 A.M. - 57, 59, 64, 65, 67, 52, 54, 61, 62, 76, 82, 83, 84, 86, 45, 85

P.M. - 86, 84

May 27 A.M. - 61, 52, 53, 54, 55, 60, 45, 46, 74, 75, 66, 86

P.M. - 18, 74, 66

May 28 A.M. - 53, 60, 55, 66, 74, 75, 87, 32, 33, 34, 35, 1

P.M. - 51, 50, 55, 87, 88, 89, 21, 33, 34, 45, 46, 35, 19, 20

May 29 A.M. - 50, 60, 62, 269, 270, 271, 277, 278, 88, 84, 35, 37, 36, 20, 19, 17, 22, 1, 18, 16

P.M. - 271, 269, 268, 258, 263, 278, 279, 280, 88, 36, 37, 16, 22

May 30 A.M. - 256, 257, 261, 264, 267, 88, 89, 280, 279, 282, 283, 285, 17, 16, 21, 2

P.M. - 261, 262, 264, 22, 13, 16

May 31 A.M. - 254, 252, 265, 259, 264, 261, 39, 40

June 1 P.M. - 285, 283, 41, 38

June 2 A.M. - 262, 261, 266, 253, 255, 285, 286, 282, 284, 275, 38, 41, 13, 16

P.M. - 254, 255, 250, 253, 266, 284, 287, 286, 36, 245, 41, 40, 72, 286, 284, 287, 276, 275, 274

June 3 A.M. - 257, 260, 264, 250, 251, 253, 266, 274, 275, 31, 30, 42, 245, 13, 219, 234, 31

P.M. - 276, 287, 288, 274, 275, 232, 29, 219, 220, 12, 218, 216, 222, 245, 246, 31

June 4 A.M. - 246, 237, 42, 232, 233, 234, 235, 236, 39, 40, 41

P.M. - 288, 289, 27, 31, 28, 44, 43, 30, 7, 6, 22, 12, 10, 16, 11

June 5 A.M. - 288, 289, 29, 44, 28, 30, 43, 28, 31, 222, 22, 14, 15, 217, 11, 10, 8

June 6 A.M. - 28, 29, 30, 44, 239, 2, 3, 7, 10, 6, 221, 222, 217, 216

P.M. - 10, 8, 222, 221, 4, 6, 5

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